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CHLORINE ENG CO LTD

14.10.87-JP-257400 (19.04.89) B01d-13/02 C07c-99 C07c-101

Aminoacid prepn. - by setting cation exchange and bipolar-membranes alternatively, feeding aminoacid alkali salt into obid. chambers, etc.

C89-071284

Prepn. of aminoacid by electrodialysis of aminoacid alkali salts comprises setting alternatively cation exchange- and bipolar-membranes of which cation side is placed toward cathode, feeding aminoacid alkali salt into chambers formed by cation side of bipolar and cation exchange-membranes, recovering corresponding aminoacid from chambers and caustic alkali from chambers formed by anion side of bipolar- and cation exchange-membranes. Pref. the electrodialysis is conducted at constant current until conversion of salt to acid reaches predetermined value, followed by operation at constant voltage.

USE/ADVANTAGE - Provides convenient regeneration of aminoacids from corresponding alkali salts with low electrolysis voltage and reduced electricity. (5pp Dwg.No.0/0)

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 PCT publication no/date

Title of invention: PRODUCTION OF AMINO ACID

Applicant: CHLORINE ENG CORP LTD

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[WHAT IS CLAIMED IS:]

[Claim 1]

In method producing amino acid by electrodialysis from alkali salt of amino acid; Production method of amino acid; wherein; The double pole membranous cation side is turned to cathode, and double pole membrane and cation-exchanger membrane are disposed in alteration, alkali salt of amino acid is supplied in chamber formed in positive ion side and cation-exchanger membrane of double pole membrane, and electrodialysis is done, caustic alkali is got from chamber formed amino acid to be suitable for in negative ion side and cation-exchanger membrane of double pole membrane by said chamber.

[Claim 2]

Production method of amino acid as claimed in item the first claims; wherein; Till the value which conversion ratio from salt of amino acid to amino acid set beforehand is reached, electrodialysis is done in constant current, electrodialysis is done by constant voltage after having reached preset value.

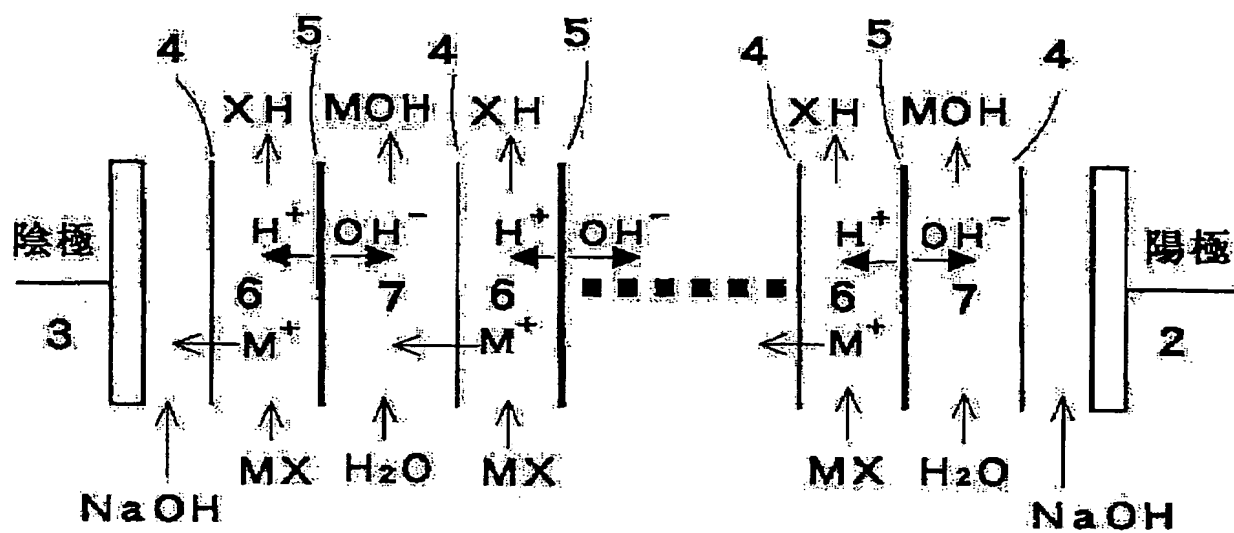
[DETAILED DESCRIPTION OF THE INVENTION]

(a field of industrial application) the present invention relates to the method that organic acid produces amino acid from alkali salt of amino acid in particular by means of electrodialysis. (prior art) amino acid produces in fermentation method or hydrolysis of nitril compound. Alkali salt of amino acid is processed in various method, and amino acid is produced so that it is in morphology of alkali salt as for the amino acid when hydrolysis is done in alkali. Method done conventionally reacts with inorganic acid of hydrochloric acid, and it is done with solution of amino acid and inorganic salt, electrolysis does alkali salt of amino acid in electrolytic cell partitioned off in the method which it makes salt of method and the amino acid which inorganic salt is separated from this solution in crystallization, and get amino acid come in contact with cation-exchanger membrane resin, and get amino acid by means of ion-exchange reaction or cation-exchanger membrane, the method which produced amino acid was done. (with the problems that invention is going to solve), in the method which roller processed by means of inorganic acid, the isolation which was complete of precipitated inorganic salt was not easy, and expenditure of inorganic acid was issue. In addition, Regeneration of ion-exchange resin operation is requirement, and it is not possible for *renzokuka* of operation to be method using ion exchange resin, and there is the issue which acid of most is of requirement in recovery of cation-exchange resin and because comparable big voltage is requirement in electrolysis with the use of cation-exchanger membrane, enough efficiency is not provided electric power cost is high and when conversion ratio from alkali salt of amino acid to acid is going to be raised, it is planted, there were problems to be massive cost of a cost of equipment of electric pole. (measure to solve problems) the present invention establishes cation-exchanger membrane in the location which is most near in cathode in double pole membrane having the character which separates aqua from in hydrogen ion and hydroxyl ion and cation-exchanger membrane to solve the problems, aqueous solution of alkali salt of amino acid is supplied in the chamber which positive ion side of double pole membrane seems to turn in cathode, and is formed in positive ion side and cation-exchanger membrane of double pole membrane in the electrodialyzer which disposed by alteration, in chamber formed in negative ion side and cation-exchanger membrane of double pole membrane, alkaline water solution of aqua or thin is supplied, and electrodialysis is done. When cation-exchanger membrane and double pole film of the same number are disposed alternately, alkali produces in positive pole side of double pole membrane or double pole membrane of the location which is most near in positive pole, but, because preferred alkali and anolyte to produce in electrodialysis are separated from, and density of anolyte not receiving effect of product by electrodialysis, cation-exchanger membrane is disposed between double pole membrane and the positive pole which are most near in positive pole, and positive pole chamber had better be established in otherwise. With progression of electrodialysis of alkali salt of amino acid, density of alkali salt of amino acid decreases, density of amino acid rises, but, when density of amino acid goes over a certain quantity, pH of solution suddenly falls. When pH of solution deteriorates, and isoelectric point is gone over, amphoteric such as for example amino acid, amino acid forms amino acid cation. As a result, It transmits in cation-exchanger membrane, and it is easy to come to transmit in side of caustic alkali, along with fall of fineness of caustic alkali, current efficiency of manufacture of amino acid goes low. On the other hand, Because conductance of amino acid is extremely small in comparison with aqueous solution of alkali salt of amino acid, electrodialysis travels, and alkali salt of amino acid decreases, because when amino acid increases, conductance in electrodialyzer falls, electric power to need in manufacture of amino acid of unit weight under break when rise of electrodialysis voltage continues electrolysis in beginning, constant current becomes massive. Figure 2 is accompanied in the case of sodium of alanine as an example of connection with pH of conversion ratio to amino acid of aqueous solution of alkali salt of amino acid and the aqueous solution, and it is shown, but, when the rate which varied from to amino acid alkali salt of conversion ratio namely original alanine goes over 90%, pH suddenly falls. Therefore, Electrodialysis is done in constant current to territory before pH suddenly deteriorates, and current efficiency deteriorates, the conversion ratio which is high without raising general electric power consumption rate by doing electrodialysis by constant voltage afterwards can be held. Electrodialysis of the first grade by constant current and electrodialysis by constant voltage afterwards the second grade, of batch target, it may be done, and process liquid of first step is introduced into sequentially secondary electrodialyzer, and it is serial, and it may be done. Amino acid provided by means of method of the present invention may combine art other than method by ion exchange resin if necessary. (action) Because, in method of the present invention, film having the function which changed aqua to hydrogen ion and hydroxyl ion was employed, because, in comparison with method by electrolysis using cation-exchanger membrane, electrolysis voltage can be largely decreased, electric power consumption rate gets possible to be decreased. In addition, Because there is not the thing that electric pole is limited to electric pole of platinum system by cation-exchanger membrane is used, and positive pole chamber and cathode chamber are established in the location which is the nearest to cathode, and doing anolyte and catholyte with alkali, because application of material of most is possible and positive pole of couple and cathode are established in positive pole chamber of both ends of electrodialyzer and cathode chamber and are preferable, a cost of equipment is cheap. Even more particularly, Amino acid to be suitable for from alkali salt of amino acid is produced without supplying acid or alkali from the outside in method of the present invention, and alkali can be reproduced. Hence: By means of hydrolysis operation puts provided alkali, and reusing, consumption figure of alkali employing in hydrolysis operation gets possible to be decreased. (embodiment) it is the thing

which figure 1 shows method of this device in when the present invention is explained in detail based on drawing more, but, positive ion side of double pole membrane is turned to cathode side, and, electrodialyzer 1, is disposed cation-exchanger membrane 4 and double pole membrane 5 of a plural number between positive pole 2 and cathode 3 by alteration. To chamber 6 of the double pole membranous cation side, alkali salt of amino acid (MX) is supplied, the alkali which was aqua or thin was added to chamber 7 of negative ion side of double pole membrane. Voluntary electrolytic solution can be used to cathode room 8 in both ends of electrodialyzer and anode chamber 9, but, when caustic alkali employing in electrodialyzer to polar regions chamber is used, it is preferable. As thus described not only it becomes easy feed facilities of electrolytic solution when electrolyte of both electrode room is done in caustic alkali, from positive pole and cathode, oxygen and hydrogen, only occurring, of special exclusion facilities as opposed to evolved gas, it does not have. On the other hand, Because application of cheap material of comparison aside from material of platinum system is possible about anode and cathode material, a cost of equipment becomes cheap. Amino acid is taken out for intermission after of continuation or electrification of appointed time from chamber 6 of the double pole membranous cation side, and alkali is taken out from chamber 7 of negative ion side of double pole membrane in similar. In addition, Figure 3 shows flow sheet of electrodialyzer enforcing method of the present invention, cathode chamber 8 and positive pole chamber 9 is coupled with each catholyte cycle tank 10 and anolyte cycle tank 11, as for chamber 6 of positive ion side of double pole membrane, sodium hydroxide cycle tank 13 is coupled with chamber 7 of negative ion side of alanine cycle tank 12 and double pole membrane. When it is possible in neither of batch type and continuous system, and it is serial, and alanine is taken out, each is serial to alanine cycle tank and sodium hydroxide cycle tank, and maneuvering of electrodialyzer dopes sodium aqueous solution of alanine and conductivity water, when alanine aqueous solution and sodium hydroxide of appointed conversion ratio are taken out, it is preferable. While each density circulates through sodium hydroxide aqueous solution of 100g/L in in sodium hydroxide aqueous solution of 80g/L and positive pole chamber and cathode chamber density in chamber of negative ion side in aqueous solution of sodium of alanine of 220g/L density in chamber of positive ion side of double pole membrane of the electrodialyzer which cation-exchanger membrane is done toward side of cathode at both ends, and disposed positive ion side of double pole membrane in alteration in double pole film of eight pieces of example 1 and nine pieces of cation-exchanger membrane, till it is to 85% conversion ratio in current density of 10A/dm^2 at early stage voltage 16V, it was energized. It turned on electricity by constant voltage of 25V afterwards for one hour. Conversion ratio to muriatic alanine of alanine was 96.5%, and average current efficiency was 86.6%, and the alanine which shifted within sodium hydroxide aqueous solution was 0.3% as sodium of alanine. In addition, The electric power consumption rate was 0.072 K W H about alanine 1g chemical equivalent. Electricity by constant current of 5A/dm^2 is done to conversion ratio 90% in current density at example 2 early stage tension 12.5V, besides what was energized by constant voltage of 20V for one hour, it was energized in condition same as superscription example 1 afterwards. The alanine that the conversion ratio to alanine shifted to 96.5%, average current efficiency 82.5%, sodium hydroxide aqueous solution was 0.3% as sodium of alanine. In addition, The electric power consumption rate was 0.059 K W H about alanine 1g chemical equivalent. Electrodialysis is done in condition same as example 1 except that it is serial, and aqueous solution of alkali salt of example 3 alanine and conductivity water were supplied in alanine cycle tank, sodium hydroxide aqueous solution cycle tank respectively, it is serial, and conversion ratio to alanine takes out sodium hydroxide aqueous solution of alanine aqueous solution and 80g/L of 85%, and, even more particularly, it is supplied in alanine cycle tank of electrodialyzer same as example 1, till conversion ratio to alanine becomes 96% with current density 3A/dm^2 , electrodialysis was done. Till conversion ratio to alanine becomes 96%, the total amount of the alanine which shifted within sodium hydroxide aqueous solution was 0.3% as sodium of alanine. In addition, The electric power consumption rate was 0.082 K W H about alanine 1g chemical equivalent. (an effect of the invention) cation-exchanger membrane is established in the location which is most near in cathode in double pole membrane having the property which separates aqua from in hydrogen ion and hydroxyl ion and cation-exchanger membrane, aqueous solution of alkali salt of amino acid is supplied in the chamber which positive ion side of double pole membrane seems to turn in cathode, and is formed in positive ion side and cation-exchanger membrane of double pole membrane in the electrodialyzer which disposed by alteration, it is the method which alkaline water solution of aqua or thin is supplied in chamber formed in negative ion side and cation-exchanger membrane of double pole membrane, and do electrodialysis, according to the current invention, amino acid and caustic alkali to be suitable for from alkali salt of amino acid can be produced in small power consumption without supplying drug from external.

[BRIEF DESCRIPTION OF DRAWINGS]

Figure 1 is figure showing an example of electrodialyzer enforcing method of the present invention. Figure 2 is figure to show connection with proportion and pH changed to amino acid of alkali salt of alanine in. Figure 3 shows flow sheet of electrodialyzer enforcing method of the present invention.



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⑭ 発明の名称 アミノ酸の製造方法

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明 細 書

1 発明の名称

アミノ酸の製造方法

2 特許請求の範囲

(1) アミノ酸のアルカリ塩から電気透析によりアミノ酸を製造する方法において、複極膜の陽イオン側を陰極に向けて、複極膜と陽イオン交換膜を交互に配置し、複極膜の陽イオン側と陽イオン交換膜で形成された室にアミノ酸のアルカリ塩を供給して電気透析し、該室から相応するアミノ酸を、複極膜の陰イオン側と陽イオン交換膜で形成された室から苛性アルカリを得ることを特徴とするアミノ酸の製造方法。

(2) アミノ酸の塩からアミノ酸への転化率があらかじめ設定した値に達するまで定電流で電気透析をし、設定値に達した後は、定電圧で電気透析をすることを特徴とする特許請求の範囲第1項に記載のアミノ酸の製造方法。

3 発明の詳細な説明

(産業上の利用分野)

本発明は、電気透析によって有機酸特にアミノ酸のアルカリ塩からアミノ酸を製造する方法に関する。

(従来技術)

アミノ酸は発酵法あるいはニトリル化合物の加水分解で製造することが行われている。加水分解をアルカリで行うとアミノ酸はアルカリ塩の形態となるために、アミノ酸のアルカリ塩を各種の方法で処理してアミノ酸を製造している。

従来行われている方法は、塩酸などの無機酸と反応させてアミノ酸と無機塩の溶液とし、この溶液から無機塩を晶析で分離してアミノ酸を得る方法や、アミノ酸の塩を陽イオン交換膜樹脂と接触させてイオン交換反応によってアミノ酸を得る方法あるいは陽イオン交換膜で仕切られた電解槽においてアミノ酸のアルカリ塩を電気分解し、アミノ酸を製造する方法が行われていた。

(発明が解決しようとする問題点)

ところが、無機酸によって処理する方法では、析出した無機塩の完全な分離は容易でなく無機酸

の消費も問題であった。

また、イオン交換樹脂を用いる方法ではイオン交換樹脂の再生工程が必要であり、運転の連続化ができないうえに、陽イオン交換樹脂の再生に多くの酸が必要である等の問題があり、また陽イオン交換膜を用いた電気分解では、比較的大きな電圧が必要であるため、電力コストが高く、またアミノ酸のアルカリ塩から酸への転化率を高めようとすると十分な効率を得られないうえ、電極などの設備費のコストが大きいという問題点があった。(問題点を解決するための手段)

本発明は、上記の問題点を解決するために、水を水素イオンと水酸イオンに分離する性質を有する複極膜と陽イオン交換膜を、陰極に最も近い位置に陽イオン交換膜を設け、複極膜の陽イオン側が陰極に向くように交互に配置した電気透析装置において、複極膜の陽イオン側と陽イオン交換膜とで形成される室にアミノ酸のアルカリ塩の水溶液を供給し、複極膜の陰イオン側と陽イオン交換膜で形成される室には、水または希薄のアルカリ

水溶液を供給して電気透析するものである。

同数の陽イオン交換膜と複極膜とを交互に配置すると陽極に最も近い位置の膜が複極膜となり、複極膜の陽極側にはアルカリが生じるが、電気透析において生じるアルカリと陽極液とを分離して陽極液の濃度が電気透析による生成物の影響を受けないようにすることが好ましいので、陽極に最も近い複極膜と陽極との間に陽イオン交換膜を配置して陽極室を別に設けるのが良い。

アミノ酸のアルカリ塩の電気透析の進行に伴い、アミノ酸のアルカリ塩の濃度が減少し、アミノ酸の濃度が上昇していくが、アミノ酸の濃度がある量を超えると、急激に溶液のpHが低下する。アミノ酸のような両性電解質は、溶液のpHが低下して等電点を越えるとアミノ酸はアミノ酸カチオンを形成する。その結果、陽イオン交換膜を透過して苛性アルカリの側に透過し易くなり、苛性アルカリの純度の低下と共に、アミノ酸の製造の電流効率が低くなる。

一方、アミノ酸の導電率はアミノ酸のアルカリ

塩の水溶液に比べて極めて小さいので、電気透析が進行してアミノ酸のアルカリ塩が減少し、アミノ酸が増加すると電気透析装置内の導電率が低下するので、電気透析電圧の上昇が起こり、定電流で電解を続けて行くと単位重量当りのアミノ酸の製造に要する電力が大きくなる。

第2図に、アミノ酸のアルカリ塩の水溶液のアミノ酸への転化率と該水溶液のpHとの関係の一例としてアラニンのナトリウム塩の場合について示すが、転化率すなわち当初のアラニンのアルカリ塩がアミノ酸に変化した割合が90%を超えると急激にpHが低下している。

したがって、pHが急激に低下し電流効率が低下する前の領域まで定電流で電気透析を行い、その後は定電圧で電気透析を行うことにより、総合的な電力原単位を上昇させずに高い転化率を維持することができる。

定電流による第一段階の電気透析と、その後の第二段階の定電圧での電気透析とをパッチ的に行っても良いし、第一段階の処理液を引続き第二の

電気透析装置に導入して連続的に行っても良い。

本発明の方法によって得られたアミノ酸は必要に応じてイオン交換樹脂による方法などの他の処理方法と組み合わせても良い。

(作用)

本発明の方法では、水を水素イオンと水酸イオンに転化する機能を有する膜を使用したので、陽イオン交換膜を使用した電気分解による方法に比べて、大幅に電解電圧を減少させることができるので、電力原単位を低下させることが可能となる。

また、陽極及び陰極に最も近い位置に陽イオン交換膜を用いて陽極室と陰極室とを設けて陽極液及び陰極液をアルカリとすることにより、電極は白金族系の電極に限定されることがないので、多くの材料の使用が可能であると共に、電気透析装置の両端の陽極室及び陰極室に一对の陽極及び陰極を設けるのみで良いので、設備費が安価である。

さらに、本発明の方法では外部から酸あるいはアルカリを供給することなしに、アミノ酸のアルカリ塩から相応するアミノ酸を製造すると共に、

アルカリを再生することができるので、得られたアルカリを加水分解工程において再使用することによって、加水分解工程で使用するアルカリの消費量を減少させることが可能となる。

(実施例)

本発明を図面に基づいてさらに詳細に説明すると第1図は、この発明の方法を示すものであるが、電気透析装置1は、陽極2と陰極3の間に複数の陽イオン交換膜4と複極膜5とを複極膜の陽イオン側を陰極側に向けて交互に配置されている。

複極膜の陽イオン側の室6には、アミノ酸のアルカリ塩(MX)を供給し、複極膜の陰イオン側の室7には、水または希薄なアルカリを加えた。電気透析装置の両端にある陰極室8及び陽極室9には任意の電解液を用いることができるが、両極室共に電気透析装置で使用している苛性アルカリを用いると良い。

このように両電極室の電解液を苛性アルカリにすると電解液の供給設備が簡単となるだけでなく、陽極及び陰極からは酸素と水素が発生するのみで

あって、発生ガスに対する特別な除外設備の必要はない。一方、陽極及び陰極材料についても白金族系の材料以外の比較的安価な材料の使用も可能であるので、設備費が安価となる。

複極膜の陽イオン側の室6からはアミノ酸を連続的あるいは、所定の時間の通電の後に間欠的に取り出すと共に、同様に複極膜の陰イオン側の室7からはアルカリを取り出す。

また第3図は、本発明の方法を実施する電気透析装置のフローシートを示し、陰極室8及び陽極室9はそれぞれ陰極液循環タンク10および陽極液循環タンク11と結合されており、複極膜の陽イオン側の室6は、アラニン循環タンク12が、また複極膜の陰イオン側の室7は水酸化ナトリウム循環タンク13が結合されている。

電気透析装置の運転は、バッチ式及び連続式のいずれでも可能であり、連続的にアラニンを取り出す場合には、アラニン循環タンク及び水酸化ナトリウム循環タンクへそれぞれ連続的にアラニンのナトリウム塩水溶液及び純水を添加し、所定の

転化率のアラニン水溶液と水酸化ナトリウムを取り出すと良い。

実施例1

8枚の複極膜と9枚の陽イオン交換膜を、複極膜の陽イオン側を陰極の側に向け、陽イオン交換膜を両端にして交互に配置した電気透析装置の複極膜の陽イオン側の室には濃度が220g/Lのアラニンのナトリウム塩の水溶液を、陰イオン側の室には濃度が80g/Lの水酸化ナトリウム水溶液を、また陽極室と陰極室にはそれぞれ濃度が100g/Lの水酸化ナトリウム水溶液を循環しながら、初期電圧18Vで10A/dm²の電流密度で転化率が85%になるまで通電をした。その後25Vの定電圧で1時間通電した。

アラニンの塩のアラニンへの転化率は、98.5%であり、平均電流効率は88.8%であり、水酸化ナトリウム水溶液中に移行したアラニンは、アラニンのナトリウム塩として0.3%であった。

また、電力原単位は、アラニン1グラム当量につき0.072KWHであった。

実施例2

初期電圧12.5Vで電流密度を5A/dm²の定電流による通電を転化率90%までおこない、その後20Vの定電圧で1時間通電したこと以外は、上記実施例1と同様の条件で通電を行った。

アラニンへの転化率は、98.5%、平均電流効率82.5%、水酸化ナトリウム水溶液に移行したアラニンは、アラニンのナトリウム塩として0.3%であった。

また、電力原単位は、アラニン1グラム当量につき0.059KWHであった。

実施例3

アラニンのアルカリ塩の水溶液及び純水をそれぞれアラニン循環タンク、水酸化ナトリウム水溶液循環タンクに連続的に供給した点を除いては、実施例1と同様の条件で電気透析をし、アラニンへの転化率が85%のアラニン水溶液と80g/Lの水酸化ナトリウム水溶液を連続的に取り出して、さらに、実施例1と同様の電気透析装置のアラニン循環タンクに供給し、電流密度3A/dm²でアラニ

ンへの転化率が98%となるまで電気透析をした。

アラニンへの転化率が98%となるまでに、水酸化ナトリウム水溶液中に移行したアラニンの総量は、アラニンのナトリウム塩として0.3%であった。また電力原単位は、アラニン1グラム当量につき0.082KWhであった。

(発明の効果)

水を水素イオンと水酸イオンに分離する性質を有する複極膜と陽イオン交換膜を、陰極に最も近い位置に陽イオン交換膜を設け、複極膜の陽イオン側が陰極に向くように交互に配置した電気透析装置において、複極膜の陽イオン側と陽イオン交換膜とで形成される室にアミノ酸のアルカリ塩の水溶液を供給し、複極膜の陰イオン側と陽イオン交換膜で形成される室には、水または希薄のアルカリ水溶液を供給して電気透析する方法である本発明によれば、外部から薬剤を供給することなしに、アミノ酸のアルカリ塩から相応するアミノ酸と苛性アルカリを小さな電力消費で製造することができる。

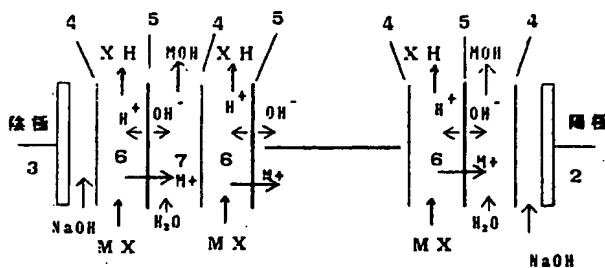
4 図面の簡単な説明

第1図は本発明の方法を実施する電気透析装置の一例を示す図である。

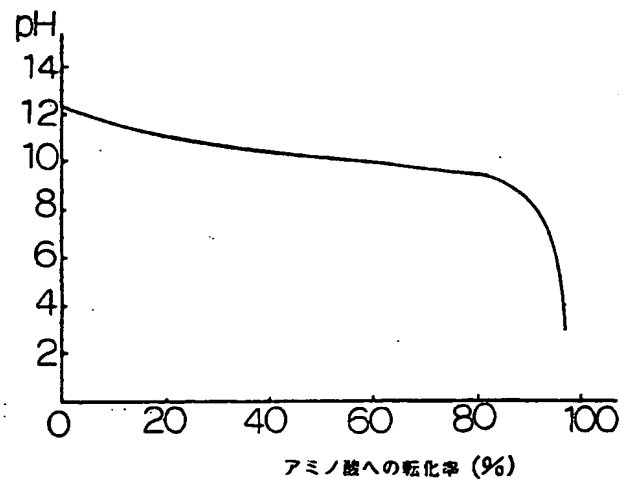
第2図はアラニンのアルカリ塩のアミノ酸へ転化した割合とpHとの関係を示す図である。

第3図は本発明の方法を実施する電気透析装置のフローシートを示す。

第1図



第2図



第3図

